

Electronic Supplementary Information

Two metal-organic frameworks sharing the same basic framework show distinct interpenetration degrees and different performances in CO₂ catalytic conversion

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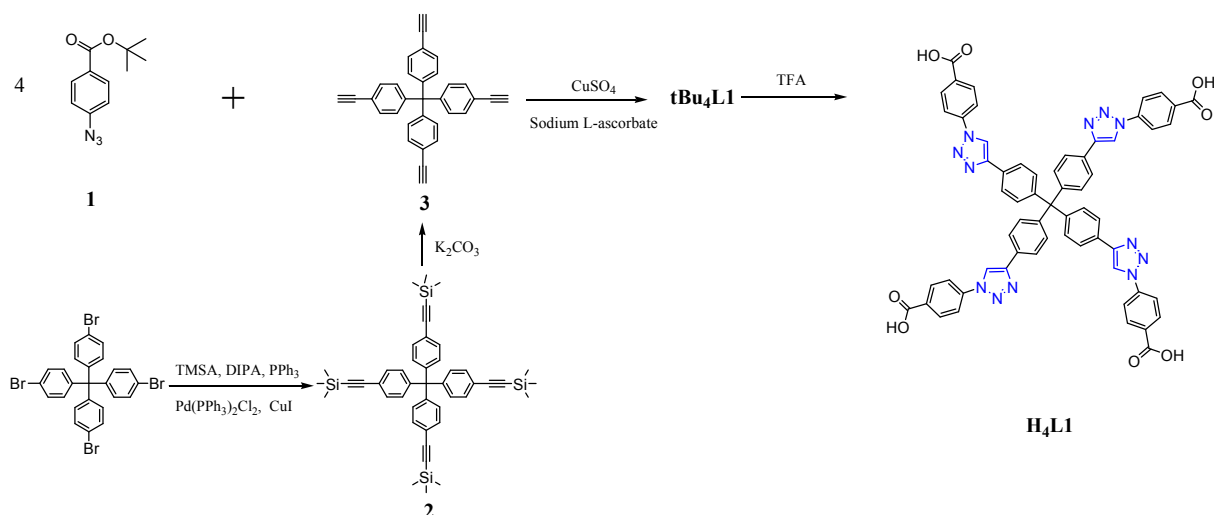
General information

All starting materials are commercially available and were used as received unless specifically mentioned: bis(triphenylphosphine)palladium(II) dichloride ($\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$, $\geq 98\%$, Sigma-Aldrich), *tert*-butyl 4-aminobenzoate ($\geq 98\%$, Sigma-Aldrich), copper(I) bromide (CuBr , $\geq 98\%$, Sigma-Aldrich), copper(II) nitrate hemi(pentahydrate) ($\text{Cu}(\text{NO}_3)_2 \cdot 2.5\text{H}_2\text{O}$, 98%, Alfa Aesar), copper(II) sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 99%, Alfa Aesar), diisopropyl amine (DIPA, $\geq 99.5\%$, Sigma-Aldrich), *N,N'*-dimehtylformamide (DMF, 99.8%, Sigma-Aldrich), hydrochloric acid (HCl, 37w%, MSR), sodium nitrite (NaNO_2 , $\geq 97\%$, Sigma-Aldrich), sodium *L*-ascorbate ($\geq 98\%$, Sigma-Aldrich), sodium azide (NaN_3 , $\geq 99.5\%$, Sigma-Aldrich), tetrakis(4-bromophenyl)methane ($\geq 95\%$, TCI), triphenylphosphine (Ph_3P , $\geq 98.5\%$, Sigma-Aldrich), and (trimethylsilyl)acetylene (TMSA, 98%, Alfa Aesar).

^1H NMR measurements were conducted on either a JEOL ECA400 NMR spectrometer or a JEOL ECA400 SL NMR spectrometer at ambient temperature. All ^1H NMR spectra were reported as chemical shift δ in units of parts per million (ppm) downfield with reference to deuterated solvent (2.50 ppm for $\text{DMSO}-d_6$ and 7.26 ppm for CDCl_3) or TMS (0.00 ppm). Multiplicities were presented as: s (singlet); d (doublet); t (triplet); and m (multiplet). Coupling constants J values were expressed in Hz and the number of protons was expressed as $n\text{H}$. ^{13}C NMR spectra were obtained from a JEOL ECA400 NMR spectrometer at ambient temperature. Spectra were reported as chemical shift δ in units of parts per million (ppm) downfield with reference to deuterated solvent (39.81 ppm for $\text{DMSO}-d_6$ or 77.16 ppm for CDCl_3-d_1). High resolution mass spectra (HRMS) or mass spectra (MS) were carried out on a Waters Q-tof Premier mass spectrometry. Melting point experiments were carried out on a SRS OptiMelt automated melting point system.

Single crystal X-ray diffraction measurements were performed on a SuperNova X-ray diffraction system from Agilent Technologies. Powder X-ray diffraction (PXRD) data were collected at 40 keV, 15 mA on a Rigaku MiniFlex 600 Benchtop diffractometer using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) over 2θ range of $2.0^\circ - 40^\circ$ at room temperature. Thermogravimetric analyses (TGA) were carried out on a TGA-Q500 thermoanalyzer with a heating rate of $10 \text{ }^\circ\text{C min}^{-1}$ under N_2 flow (60 mL/min) from room temperature to $700 \text{ }^\circ\text{C}$. Gas sorption analyses were conducted using Quantachrome Instruments Autosorb-iQ (Boynton Beach, Florida USA) with extra-high pure gases.

Experimental section



Scheme S1. Synthetic route for the preparation of ligand **H₄L1**.

tert-Butyl 4-azidobenzoate (1). Compound **1** was synthesized in accordance to literature-reported method with slight modifications.^{S1,S2} *tert*-Butyl 4-aminobenzoate (1.93 g, 10 mmol) was added into a mixture of MeOH (60 mL) and HCl (0.5 M, 60 mL) at 0 °C. Subsequently, a water solution of NaNO₂ (2.1 g, 30 mmol in 10 mL H₂O) was slowly added into the above mixture, and the resulted mixture solution was stirred at 0 °C for 1.5 h. Then, a solution of NaN₃ (1.95 g, 30 mmol in 10 mL H₂O) was slowly poured into the mixture solution at 0 °C with stirring for 2 hours, followed by extraction with CH₂Cl₂ (50 mL × 2). The combined organic layers were dried over Na₂SO₄ and the solvent was evaporated under vacuum. The crude product was purified by silica gel flash column chromatography (hexane/CH₂Cl₂, 100/20) to give compound **1** as a yellow liquid (2.11 g, 9.63 mmol, yield 96%). ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 8.8 Hz, 2H), 7.03 (d, *J* = 8.8 Hz, 2H), 1.57 (s, 9H). ¹³C NMR (126 MHz, CDCl₃) δ 165.1, 144.3, 131.3, 128.7, 118.7, 81.3, 28.3. FTIR (KBr) ν/cm^{-1} : 2978.1(m), 2931.8(m), 2125.6(s), 2090.8(s), 1712.8(s), 1604.8(s), 1504.5(m), 1396.5(m), 1288.5(s), 1257.6(m), 1165.0(s), 1130.3(s), 1111.0(s), 1014.6(w), 852.5(m), 767.7(m), 690.5(w). ESI-TOF-HRMS: *m/z* calcd for C₁₁H₁₄N₃O₂: 220.1086, found: 220.1081 [M+H]⁺.

Tetrakis(4((trimethylsilyl)ethynyl)phenyl)methane (2). Compound **2** was synthesized according to literature-reported method with slight modifications.^{S1,S3} Diisopropyl amine (15 mL) and trimethylsilyl acetylene (3.4 mL, 2.36 g, 2.40 mmol) were injected into a dried THF solution (50 mL) containing tetrakis(4-bromophenyl)methane (1.50 g, 2.36 mmol) and triphenylphosphine (62.4 mg, 0.24 mmol) under N₂ environment. Subsequently, bis(triphenylphosphine)palladium(II) dichloride (83.5 mg, 0.12 mmol) and copper(I) bromide (22.7 mg, 0.16 mmol) were added into the mixture solution under N₂ environment, and the resulted solution was subjected to reflux at 60°C for

20 hours. After cooling down to room temperature naturally, the insoluble substance was filtered out followed by CH₂Cl₂ washing. The filtrate was then washed by water and then extracted with CH₂Cl₂ (100 mL × 2). The combined organic layer was dried over Na₂SO₄ and the solvent was evaporated under vacuum. The crude product was purified by flash column chromatography on silica gel using hexane as eluent to give compound **2** as a white solid (1.28 g, 1.81 mmol, yield: 77%, melting point: 343-344 °C). ¹H NMR (400 MHz, CDCl₃): δ 7.32 (d, *J* = 8.5 Hz, 8H), 7.03 (d, *J* = 8.5 Hz, 8H), 0.23 (s, 36H). ¹³C NMR (400MHz, CDCl₃): 146.1, 131.4, 130.8, 121.3, 104.7, 94.8, 64.8, 0.0. FTIR (KBr) ν/cm⁻¹: 3448.7(m), 2958.8(m), 2924.1(w), 2852.7(w), 2158.4(s) 1496.8(s), 1406.1(w), 1249.9(s), 1186.2(m), 1018.4(m), 866.0(s), 842.9(s), 810.1(s), 760.0(s), 700.2(w), 653.9(m), 640.4(m), 597.9(w). ESI-TOF-HRMS: *m/z* calculated for Mr of C₄₅H₅₃Si₄: 705.3224, found: 705.3229 [M + H]⁺.

Tetrakis(4-ethynylphenyl)methane (3). Potassium carbonate (K₂CO₃, 1.38 g, 10.00 mmol) was added into a DCM/MeOH (30 mL/30 mL) solution of compound **2** (1.2 g, 1.70 mmol), and then the mixture was stirred at room temperature overnight. The mixture solution was poured into water (50 mL) and extracted by CH₂Cl₂ (50 mL × 2). The combined organic layer was dried over Na₂SO₄ and the solvent was evaporated under vacuum. The crude product was purified by silica gel flash column chromatography (hexane/CH₂Cl₂, 100/10) to give **3** as a white solid (0.64 g, 1.54 mmol, yield: 90%, melting point: 333-334 °C). ¹H NMR (400MHz, CDCl₃): 7.38 (d, *J* = 8.2 Hz, 8H), 7.11 (d, *J* = 8.2 Hz, 8H), 3.06 (s, 4H). ¹³C NMR (400Hz, CDCl₃): 146.3, 131.7, 130.8, 120.4, 83.3, 77.7, 64.9. FTIR (KBr) ν/cm⁻¹: 3282.8(s), 3030.2(w), 2358.9(w), 2328.1(w), 2110.1(w), 1930.7(w), 1602.9(w), 1558.5(w), 1496.8(s), 1400.3(m), 1253.7(m), 1018.4(m), 825.5(s), 667.4(m), 640.4(m), 569.0(m), 559.4(m). ESI-TOF-HRMS: *m/z* calculated for Mr of C₃₃H₂₁: 417.1643, found: 417.1633 [M + H]⁺.

Tetra-(tert-butyl) 4,4',4'',4'''-((methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1*H*- 1,2,3-triazole-4,1-diyl))tetrabenzoate ('Bu₄L1). 'Bu₄L1 was synthesized in accordance to literature-reported method with slight modifications.^{S1,S2} Above synthesized **1** (1.05 g, 4.80 mmol) and **3** (0.42 g, 1.00 mmol) were added into a mixture of THF/H₂O (150 mL/50 mL). Then, CuSO₄·5H₂O (0.10 g, 0.40 mmol, 10 mL H₂O) and excess sodium *L*-ascorbate (0.5 g, 2.5 mmol, 10 mL H₂O) were added into the solution, and the mixture solution was stirred at 50 °C under N₂ for 3 days. The mixture solution was poured into water (50 mL) and then extracted by CH₂Cl₂ (80 mL × 2). The combined organic layer was dried over Na₂SO₄ and the solvent was evaporated under vacuum. The crude product was purified by silica gel flash column chromatography (CH₂Cl₂/EtOAc, 100/2) to give compound 'Bu₄L1 as a light yellow solid (1.18 g, 0.91 mmol, yield: 91%, became black before

melting at above 270 °C). ¹H NMR (400MHz, DMSO-*d*₆): 9.39 (s, 4H), 8.13 (d, *J* = 9.1 Hz, 8H), 8.10 (t, *J* = 9.1 Hz, 4H), 7.96 (d, *J* = 8.3 Hz, 8H), 7.48 (d, *J* = 8.3 Hz, 8H), 1.57 (s, 36H). ¹³C NMR (400MHz, DMSO-*d*₆): 164.6, 147.8, 146.8, 140.0, 131.6, 131.6, 131.4, 128.5, 125.7, 120.3, 120.2, 81.8, 64.9, 28.3. FTIR (KBr) ν/cm^{-1} : 3456.4(m), 2976.2(m), 2931.8(m), 1708.9(s), 1608.6(s), 1558.5(m), 1518.0(m), 1489.1(m), 1440.8(m), 1408.0(m), 1392.6(m), 1369.5(m), 1294.2(s), 1255.7(m), 1228.7(m), 1163.1(s), 1120.6(s), 1031.9(m), 1018.4(m), 993.3(m), 858.3(m), 846.8(m), 829.4(m), 792.7(w), 769.6(m), 692.4(w). ESI-TOF-HRMS: *m/z* calculated for Mr of C₇₇H₇₃N₁₂O₈: 1293.5674, found: 1293.5679[M + H]⁺.

4,4',4'',4'''-((Methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1*H*-1,2,3-tri-azole-4,1-diyl)) tetra benzoic acid (H₄L1). To a CH₂Cl₂ (DCM) solution (20 mL) of the compound 'Bu₄L1 (1.10 g, 0.85 mmol), 10 mL trifluoroacetic acid (TFA) was added. The mixture solution was stirred at room temperature overnight, obtaining white precipitates. After filtration, DCM was removed under reduced pressure, and the obtained materials were dried at 100 °C overnight to afford the compound H₄L1 (0.84 g, 0.79 mmol, yield 93%, became black before melting above 270 °C) as white solid. ¹H NMR (400MHz, DMSO-*d*₆): 13.24 (s, 4H), 9.41 (s, 4H), 8.19 (d, *J* = 8.6 Hz, 8H), 8.12 (t, *J* = 8.6 Hz, 4H), 7.97 (d, *J* = 8.2 Hz, 8H), 7.49 (d, *J* = 8.2 Hz, 8H). ¹³C NMR (400MHz, DMSO-*d*₆): 166.9, 147.8, 146.8, 140.1, 131.7, 131.6, 131.2, 128.4, 125.7, 120.3, 120.2, 64.9. FTIR (KBr) ν/cm^{-1} : 3456.4(s), 2924.1(m), 1703.1(s), 1608.6(s), 1558.5(w), 1518.0(w), 1491.0(w), 1444.7(w), 1408.0(m), 1313.5(w), 1230.6(s), 1172.7(w), 1118.7(w), 1051.2(m), 995.3(m), 858.3(w), 827.5(w), 796.6(m), 773.5(m), 692.4(w). ESI-TOF-HRMS: *m/z* calculated for Mr of C₆₁H₄₁N₁₂O₈: 1069.3170, found: 1069.3156[M + H]⁺.

MOF 1. Compound H₄L1 (10 mg, 0.009 mmol) and Cu(NO₃)₂·2.5H₂O (24 mg, 0.10 mmol) were dissolved in a *N,N'*-dimethylformamide (DMF, 10 mL) solution. Then, the mixture solution was placed in a tightly capped 20 mL vial after adding drops of nitric acid, which was heated in an oven at 80°C for 3 days. The blue block crystals were collected after cooling the sample down to room temperature. The crystals were washed by fresh DMF for three times and dried naturally at room temperature to yield **1** (Yield: 18.9 mg weighted after drying naturally).

MOF 2. Compound H₄L1 (10 mg, 0.009 mmol) and Cu(NO₃)₂·2.5H₂O (24 mg, 0.10 mmol) were dissolved in a solution consisting of DMF (8 mL), ethanol (1 mL) and H₂O (1 mL). Then, the mixture solution was placed in a tightly capped 20 mL vial after adding drops of nitric acid, which was heated in an oven at 80°C for 3 days. The blue block crystals were collected after cooling the

sample down to room temperature. The crystals were washed by fresh DMF for three times and dried naturally at room temperature to yield **1** (Yield: 14.2 mg weighted after drying naturally).

X-ray crystallography. The single crystals of **1** and **2** suitable for X-ray analysis were picked up under a microscope from synthesized solution and then sealed in a glass tube immediately with DMF solvent and mounted. The diffraction data were collected at 293 K using graphite-monochromated Cu- $K\alpha$ radiation ($\lambda = 1.54178$ Å) on a SuperNova X-ray Diffraction System from Agilent Technologies. Data reduction and empirical absorption correction were applied with CrysAlisPro.^{S4} The structures were solved by the direct methods using the SHELXS program of the SHELXTL package and refined by full-matrix least-squares analyses on F^2 (SHELXTL-2014).^{S5} Metal atoms in each complex were located from the E -maps, and other non-hydrogen atoms were located in successive difference Fourier syntheses. The hydrogen atoms of the ligand were generated theoretically onto the specific atoms and refined isotropically with fixed thermal factors. It was impossible to see clear electron-density peaks in difference maps corresponding to acceptable locations for the H atoms bonded to the oxygen (O5) atoms of the coordinated water molecules in both structures. Thus, the refinements were completed by specifying the nearest Q peaks as H atoms and fixing these water H atoms in the right models. The isolated solvent molecules in the MOF are highly disordered and cannot be modeled, thus the SQUEEZE routine of PLATON was applied to remove the contributions of the solvent molecules to the scattering.^{S6} Further details of crystallographic data and structural analysis are summarized in Table S1. CCDC 1523578 and 1523579 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table S1. Crystallographic data and structure refinement summary for MOFs **1** and **2**.

	1	2
Formula after squeeze	C ₆₁ H ₄₀ N ₁₂ O ₁₀ Cu ₂	C ₆₁ H ₄₀ N ₁₂ O ₁₀ Cu ₂
Formula with isolated solvents	{Cu ₂ (C ₅₇ H ₃₆ N ₁₂)(COO) ₄ }(H ₂ O) ₂ ·22(C ₃ H ₇ NO)}	{Cu ₂ ((C ₅₇ H ₃₆ N ₁₂)(COO) ₄)(H ₂ O) ₂ ·7(C ₃ H ₇ NO)}
Mr after squeeze	1228.13	1228.13
Mr with isolated solvents	2836.11	1739.76
Temperature (K)	293(3)	293(3)
Wavelength	1.54178 Å	1.54178 Å
Crystal system	Tetragonal	Tetragonal
Space group	<i>P4₂2₁2</i>	<i>P4₂2₁2</i>
a,b,c / Å $\alpha, \beta, \gamma / ^\circ$	a = 22.8050(5) Å, c = 44.0727(12) Å	a = 16.0604(4) Å, c = 44.1170(11) Å
V, Å ³	22920.8(12)	11379.3(5)
Z	4	4
F(000) after squeeze	2512	2512
F(000) with isolated solvents	3391	2794
D _{calc} (g·cm ⁻³)	0.356	0.717
D _{calc} with isolated solvents (g·cm ⁻³)	0.822	1.016
Crystal size (mm)	0.166 × 0.148 × 0.144	0.182 × 0.112 × 0.106
Theta range (deg)	3.40 to 73.33	3.89 to 73.16
Reflections unique/collected	6222/30623	5583/11062
Flack parameter	0.20(5)	0.08(5)
R _{int}	0.0935	0.0361
Goodness-of-fit on F ²	1.074	1.057

Figures

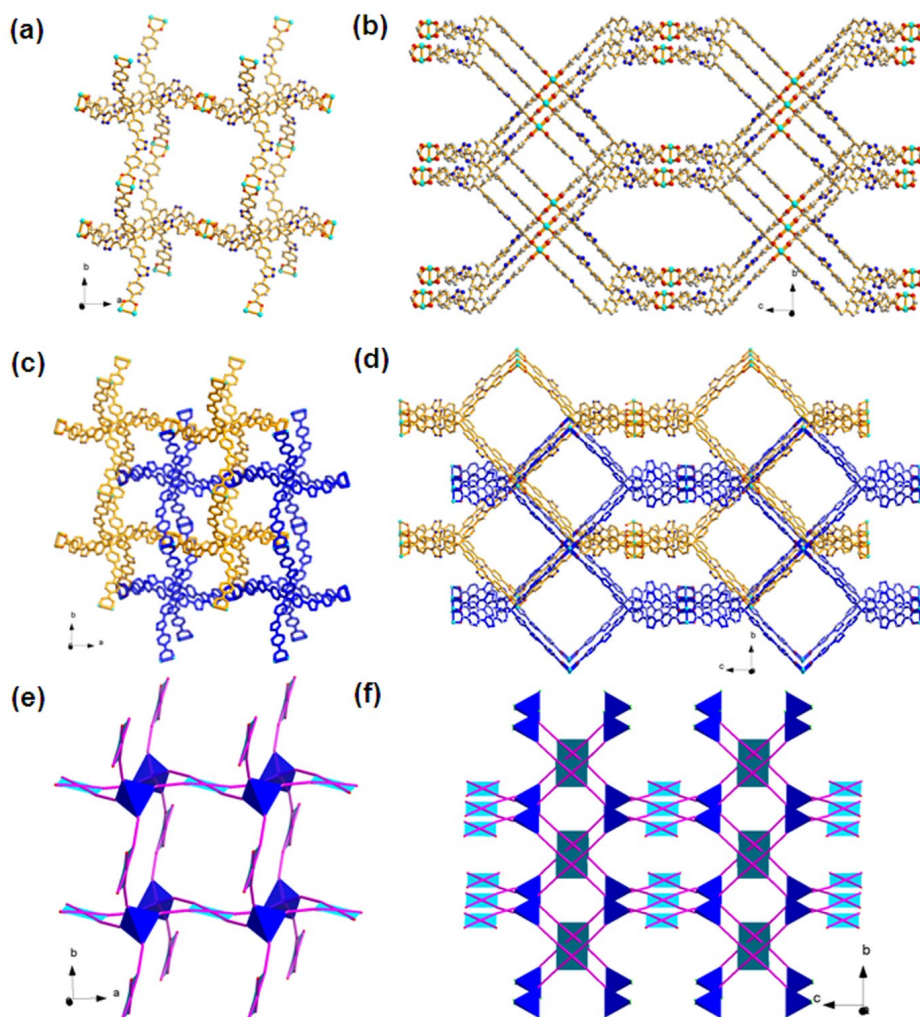


Fig. S1. Perspective view of basic 3D porous framework for **1** (a) along c -axis and (b) along a -axis; perspective view of 2-fold interpenetrated frameworks in **1** (c) along c -axis and (d) along a -axis; illustration of the “PtS”-type topological network of **1** showing the combination of planar tetratopic paddlewheel Cu_2 clusters and tetrahedral tetratopic **L1** linkers.

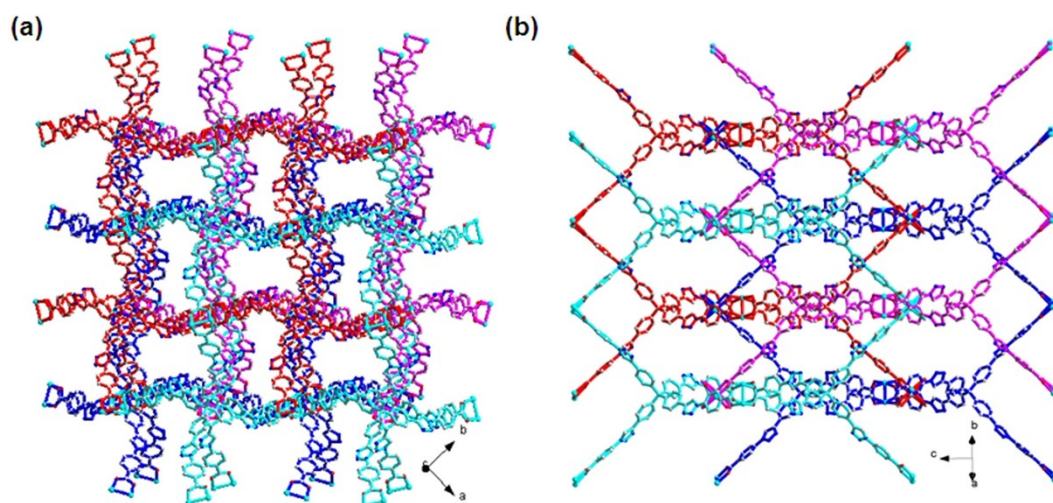


Fig. S2. Perspective view of 4-fold interpenetrated frameworks in **2** (a) along c -axis and (b) along (110)-axis.

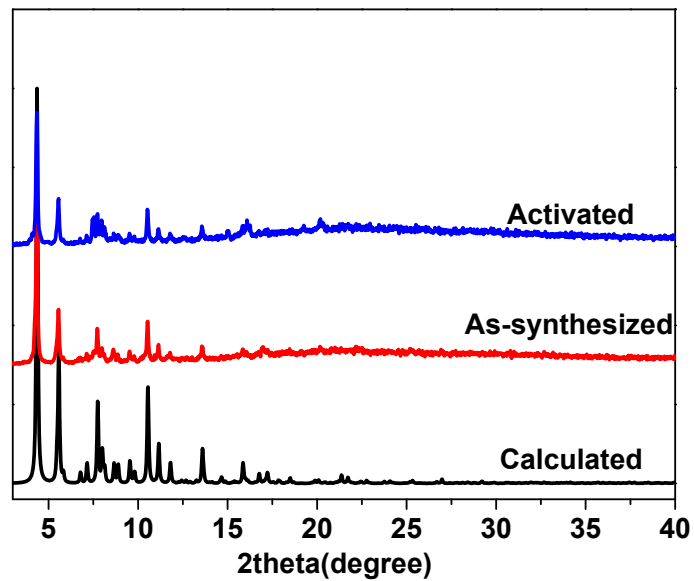


Fig. S3. PXRD patterns of **1**, indicating that the framework was retained after the activation process.

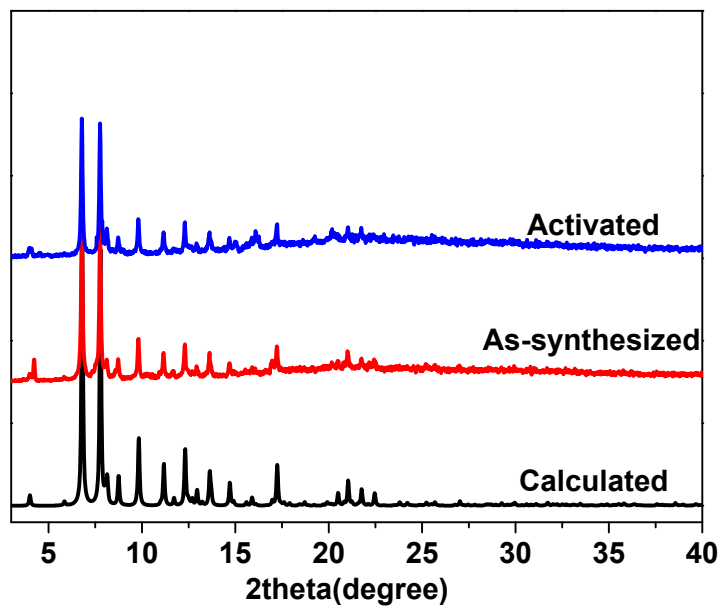


Fig. S4. PXRD patterns of **2**, indicating that the framework was retained after the activation process.

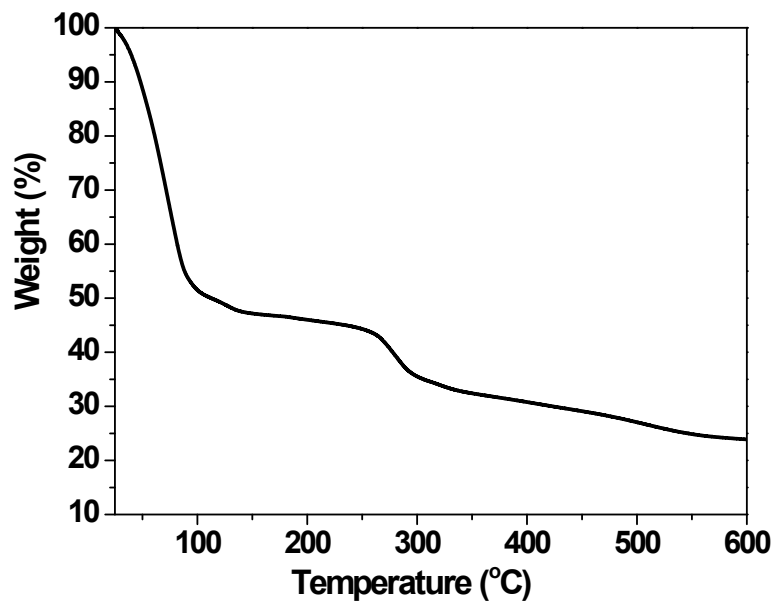


Fig. S5. TGA plot of as-synthesized **1**, indicating that the framework was stable up to ~260 °C. The initial sharp weight loss can be attributed to the weight loss of solvents (water and DMF) in pores of the framework.

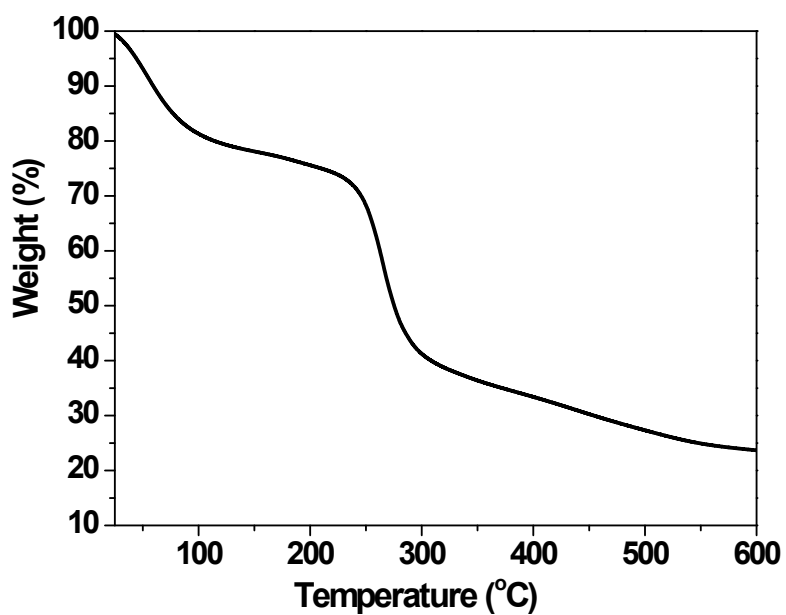


Fig. S6. TGA plot of as-synthesized **2**, indicating that the framework was stable up to ~250 °C. The initial sharp weight loss can be attributed to the weight loss of solvents (water and DMF) in pores of the framework.

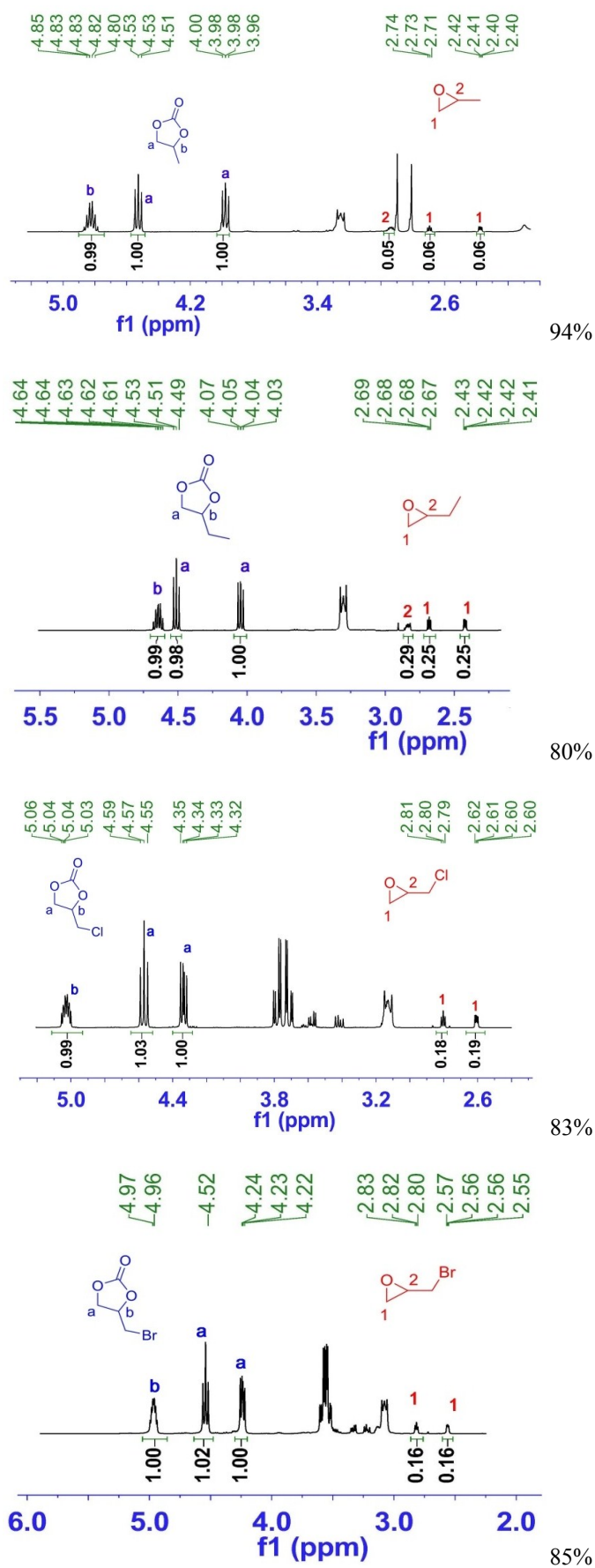


Fig. S7. ^1H NMR spectra for the cyclic carbonates (with corresponding yield) produced from the CO_2 cycloaddition with related epoxides catalyzed by **1**.

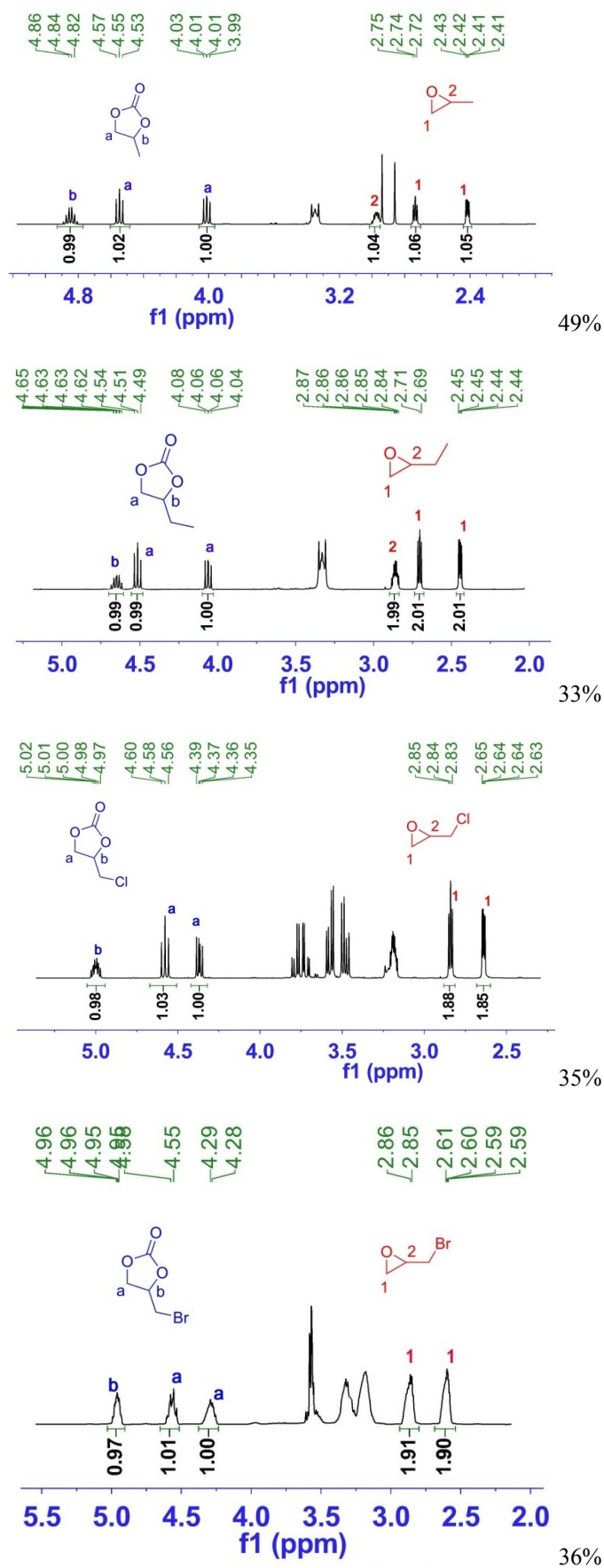


Fig. S8. ¹H NMR spectra for the cyclic carbonates (with corresponding yield) produced from the CO₂ cycloaddition with related epoxides catalyzed by **2**.

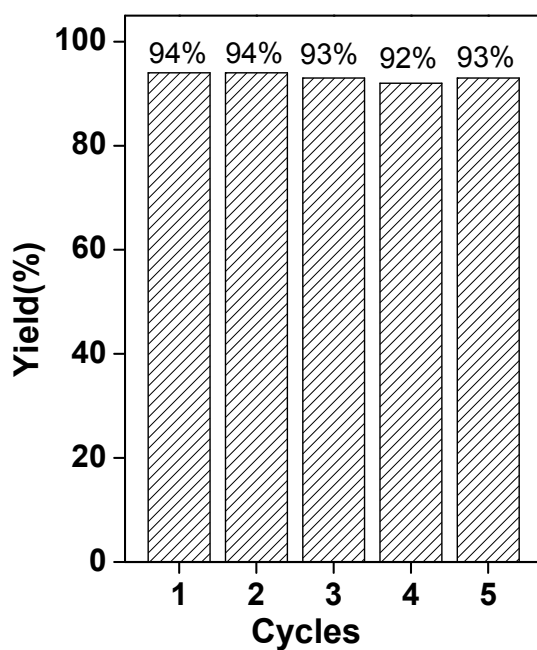


Fig. S9. Recyclability of **1** in five runs for catalytic CO₂ cycloaddition with propylene oxide to produce propylene carbonate.

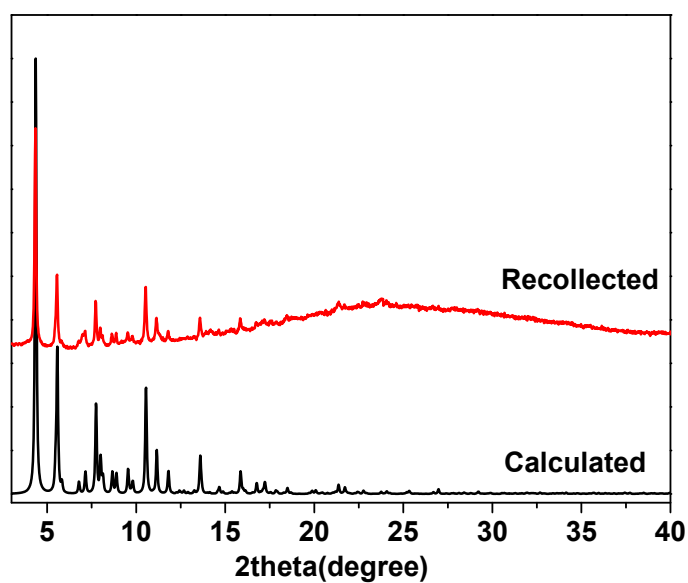
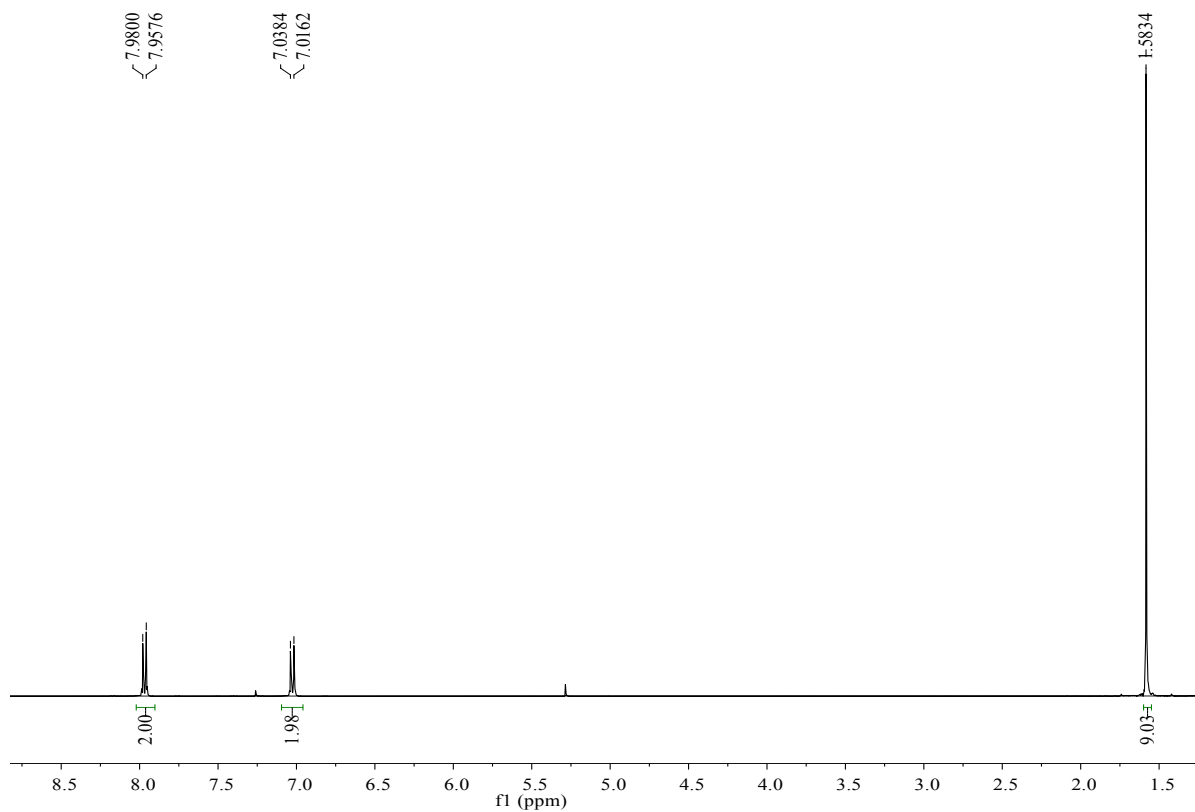
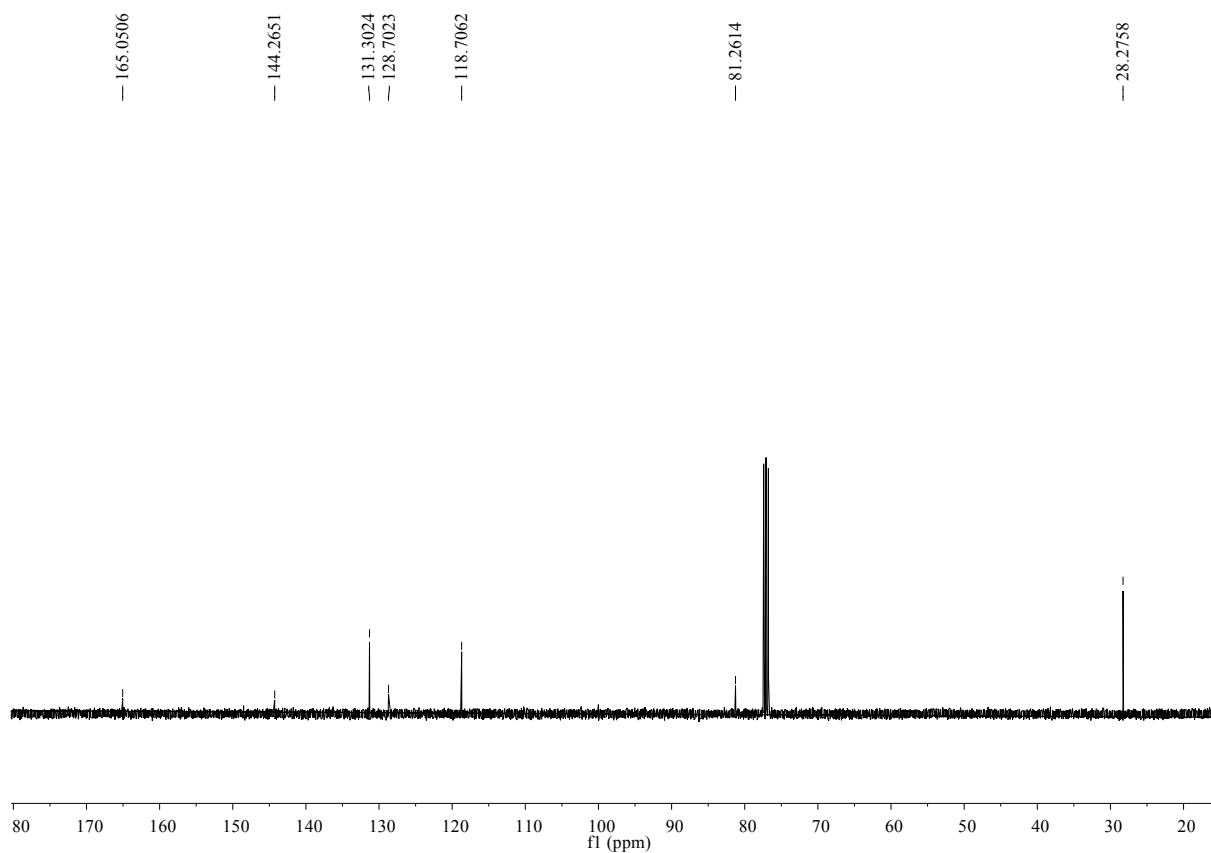


Fig. S10. PXRD patterns of **1** recollected after catalysis reactions and calculated from the crystal data, indicating that the framework was retained after the catalysis reactions.

Characterization spectra



¹H NMR spectrum of *tert*-butyl 4-azidobenzoate in CDCl₃ (trace DCM was observed).



¹³C NMR spectrum of *tert*-butyl 4-azidobenzoate in CDCl₃.

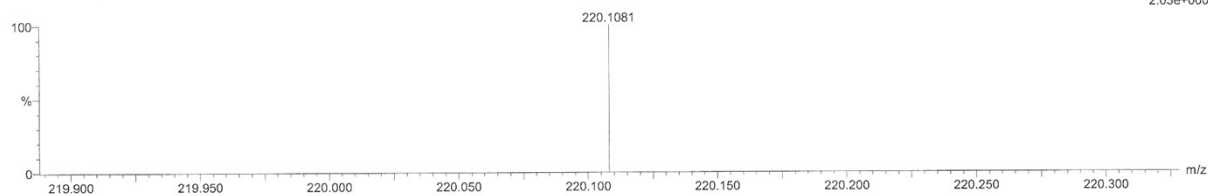
Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
 2 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

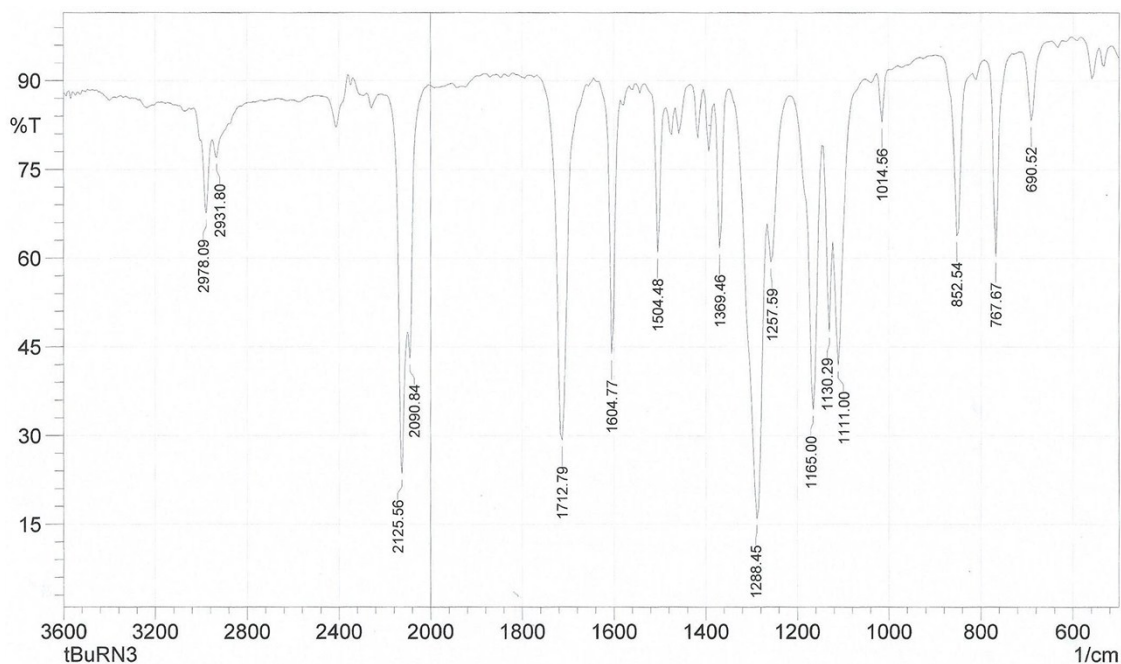
Elements Used:
 C: 11-11 H: 12-15 N: 3-3 O: 2-2 Na: 0-1
 C₁₁H₁₃N₃O₂
 tBuLR 59 (1.311)

1: TOF MS ES+
 2.03e+000

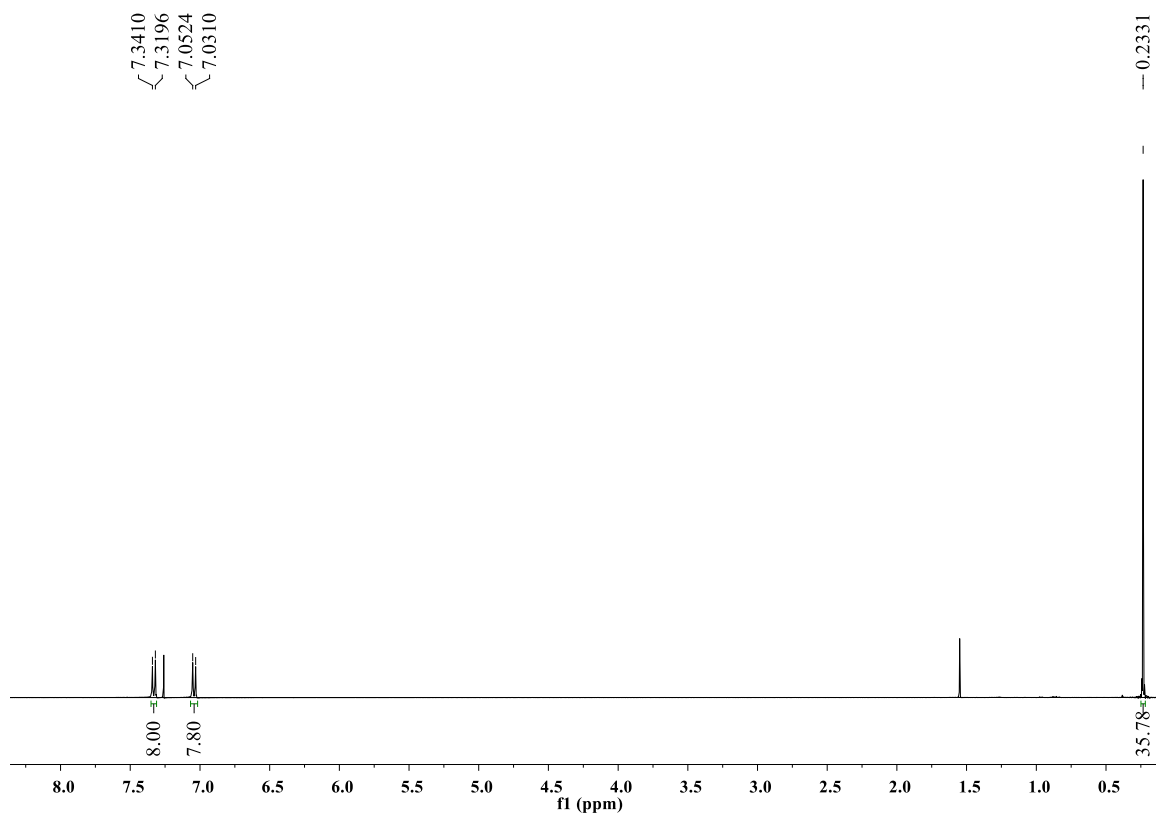


Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
220.1081	220.1086	-0.5	-2.3	6.5	12.0	0.0	C11 H14 N3 O2

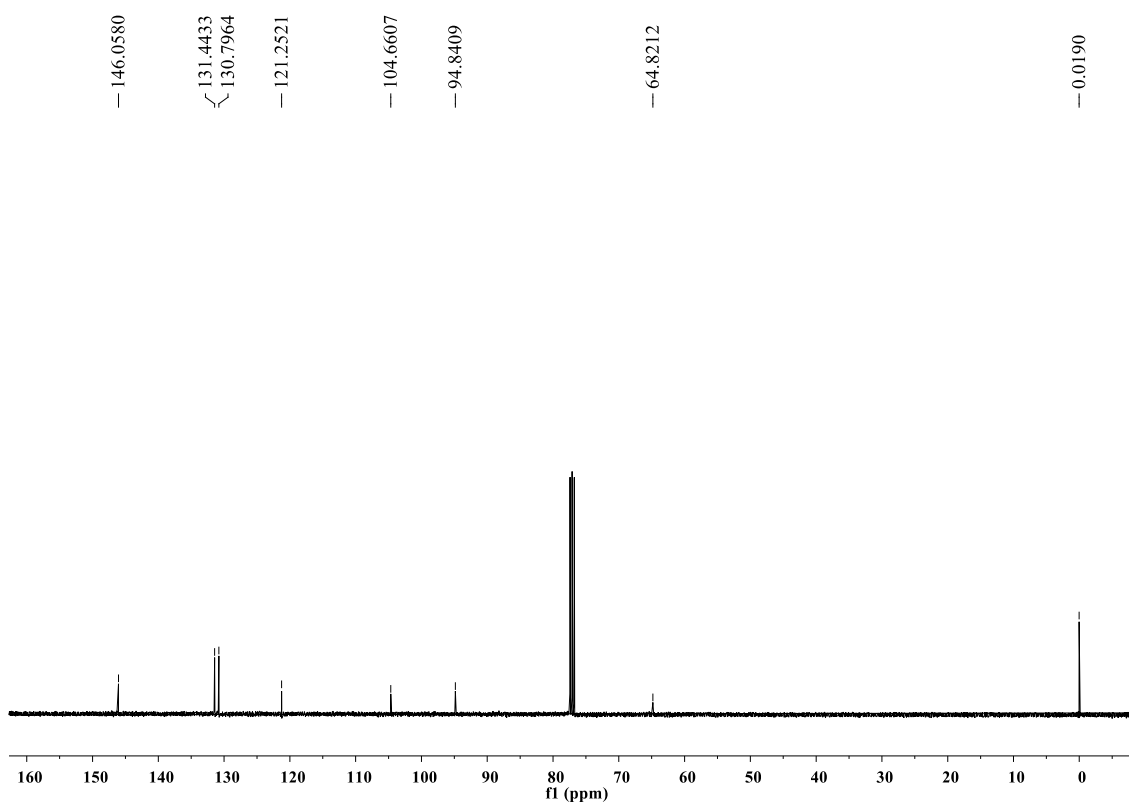
HRMS spectrum of *tert*-butyl 4-azidobenzoate



FT-IR spectrum of *tert*-butyl 4-azidobenzoate



¹H NMR spectrum of tetrakis(4((trimethylsilyl)ethynyl)phenyl)methane in CDCl₃.



¹³C NMR spectrum of tetrakis(4((trimethylsilyl)ethynyl)phenyl)methane in CDCl₃.

Elemental Composition Report

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

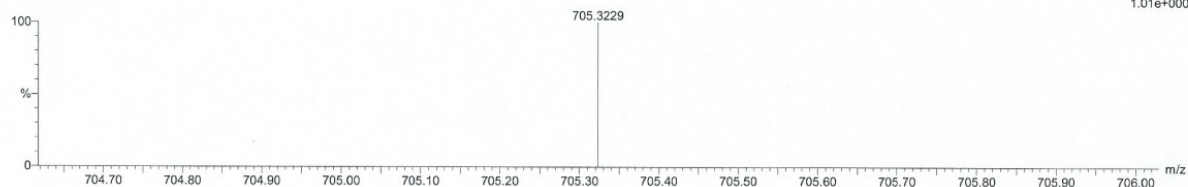
1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 45-45 H: 52-54 Si: 4-4

C₄₅H₅₂Si₄

lpzT4Si 6 (0.138)



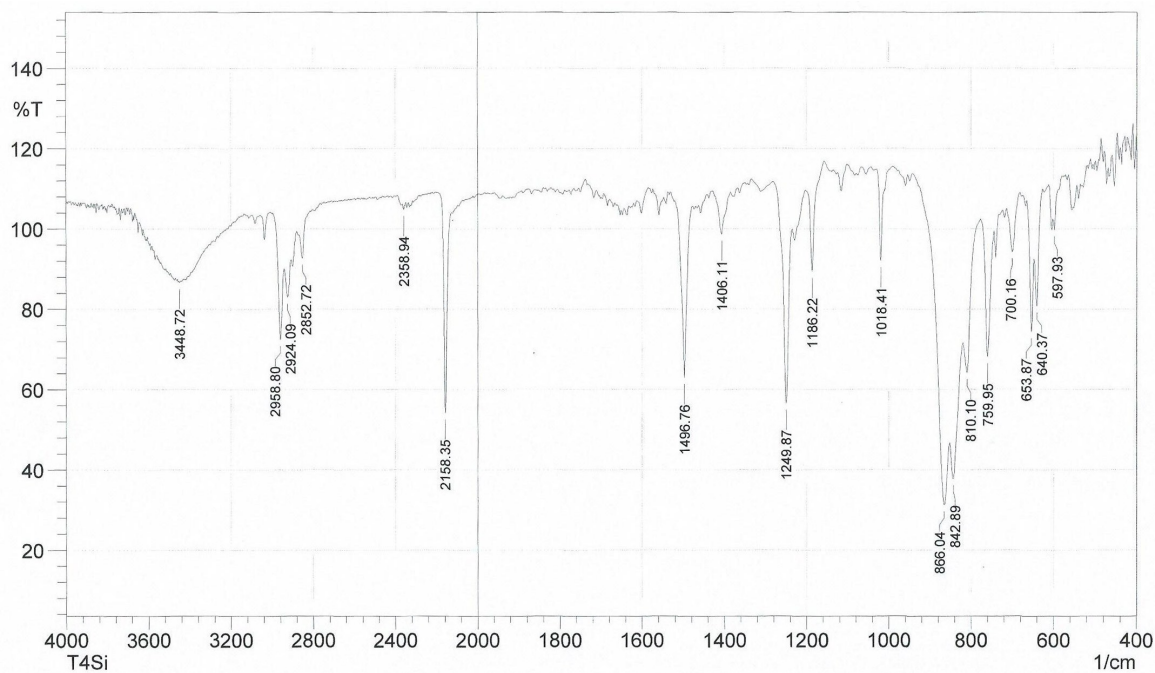
1: TOF MS ES+
1.01e+000

Minimum:

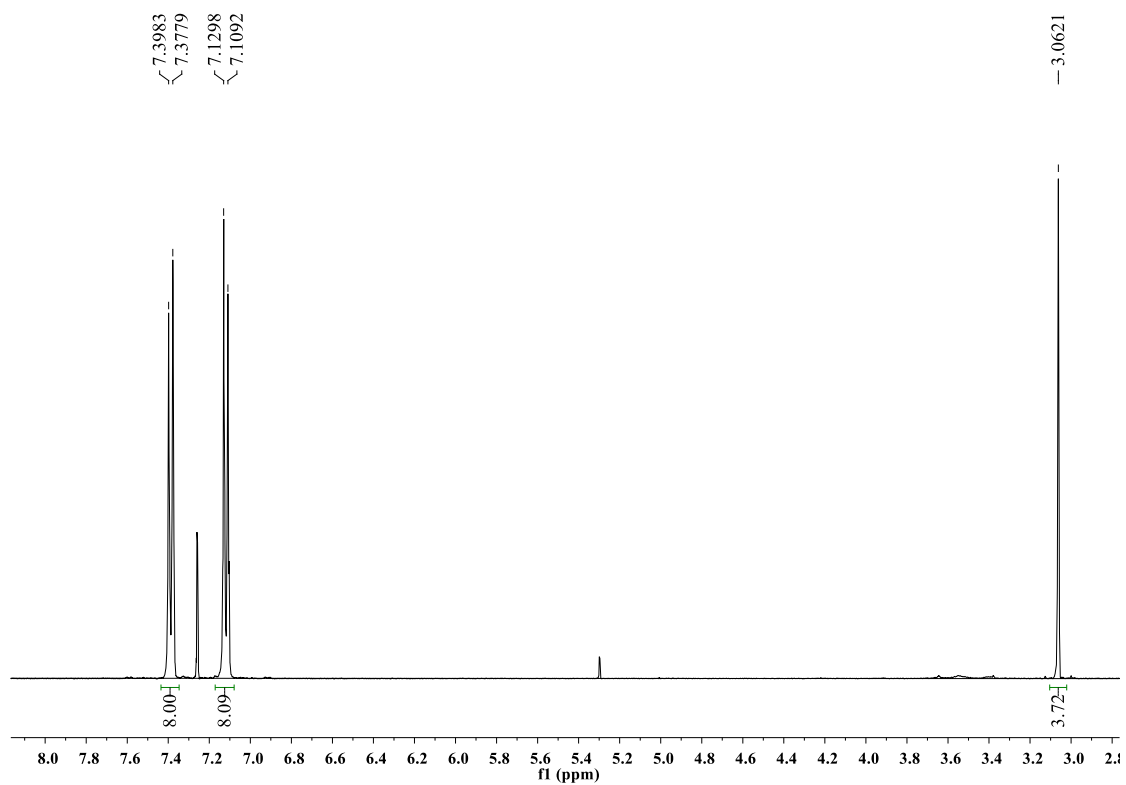
Maximum: 5.0 20.0 -1.5 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
705.3229	705.3224	0.5	0.7	23.5	12.2	0.0	C ₄₅ H ₅₃ Si ₄

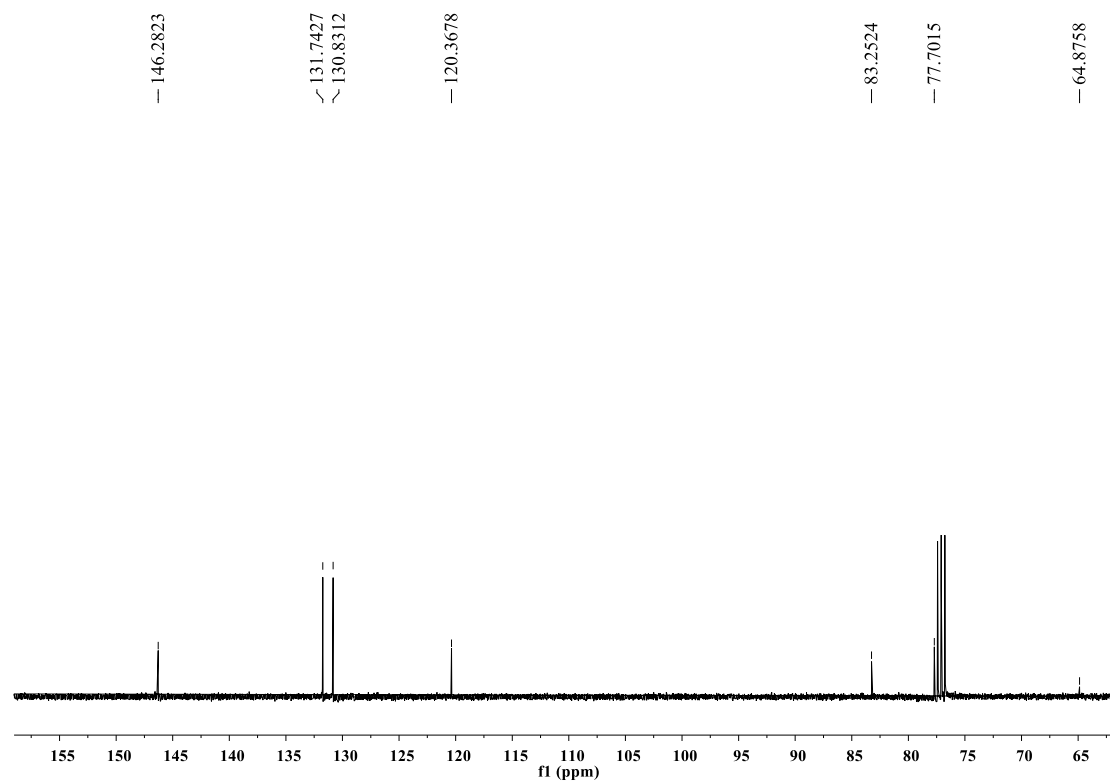
HRMS spectrum of tetrakis(4((trimethylsilyl)ethynyl)phenyl)methane



FT-IR spectrum of tetrakis(4((trimethylsilyl)ethynyl)phenyl)methane



^1H NMR spectrum of tetrakis(4-ethynylphenyl)methane in CDCl_3 (trace DCM was observed).



^{13}C NMR spectrum of tetrakis(4-ethynylphenyl)methane in CDCl_3

Elemental Composition Report

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

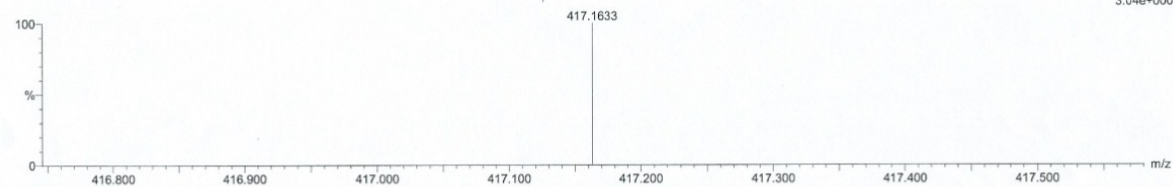
Elements Used:

C: 33-33 H: 18-22

C33H20

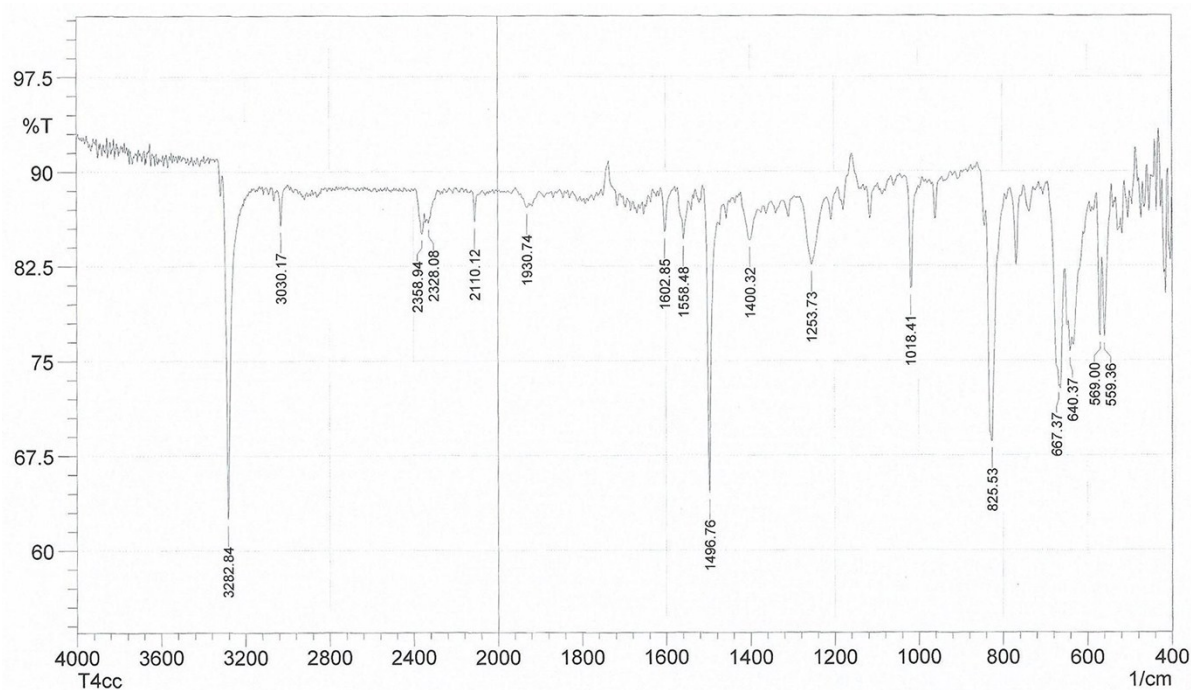
lpzT4CC 4 (0.101)

1: TOF MS ES+
3.04e+000

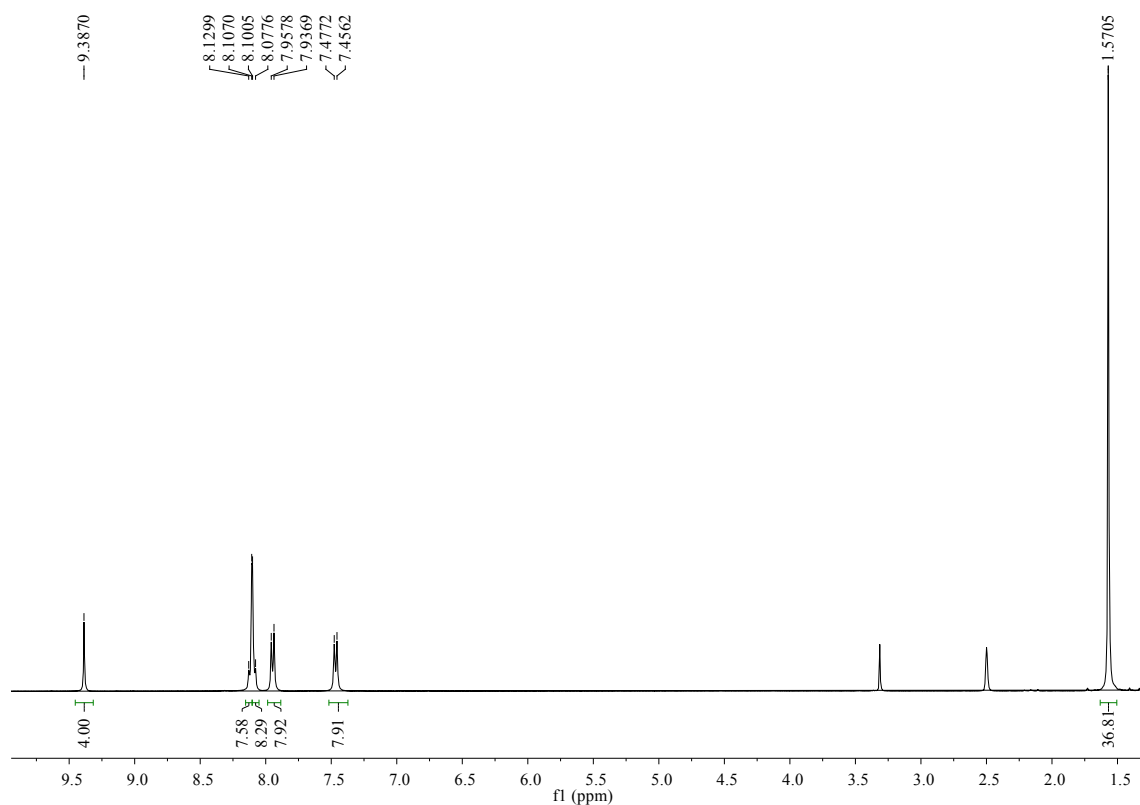


Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
417.1633	417.1643	-1.0	-2.4	23.5	11.5	0.0	C33 H21

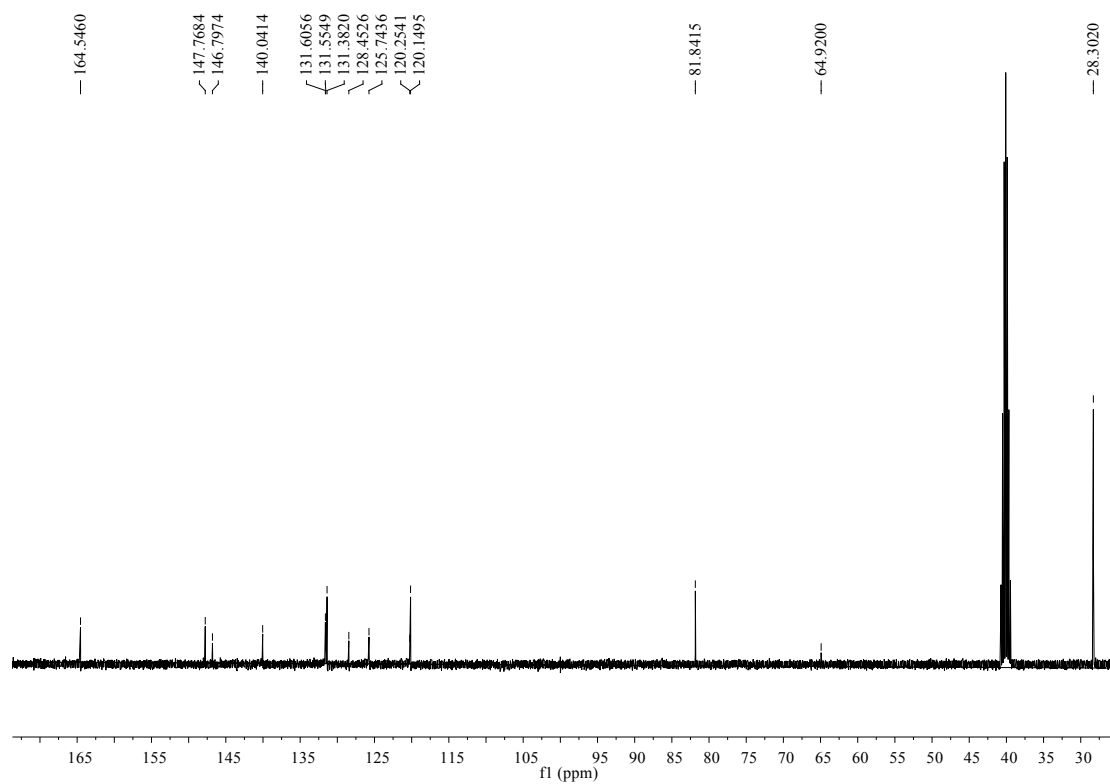
HRMS spectrum of tetrakis(4-ethynylphenyl)methane



FT-IR spectrum of tetrakis(4-ethynylphenyl)methane



^1H NMR spectrum of tetra-(*tert*-butyl) 4,4',4'',4'''-((methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1*H*- 1,2,3-triazole-4,1-diyl))tetrabenzoate in $\text{DMSO-}d_6$.



^{13}C NMR spectrum of tetra-(*tert*-butyl) 4,4',4'',4'''-((methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1*H*- 1,2,3-triazole-4,1-diyl))tetrabenzoate in $\text{DMSO-}d_6$.

Elemental Composition Report

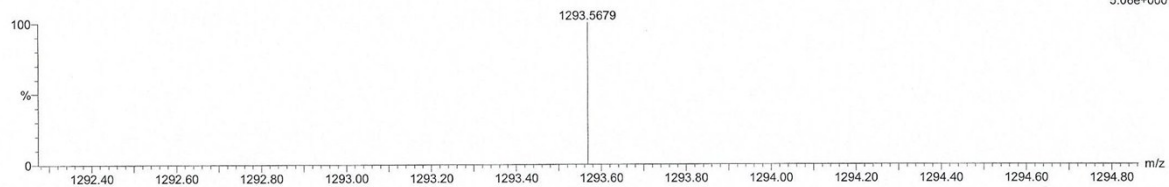
Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
 1 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

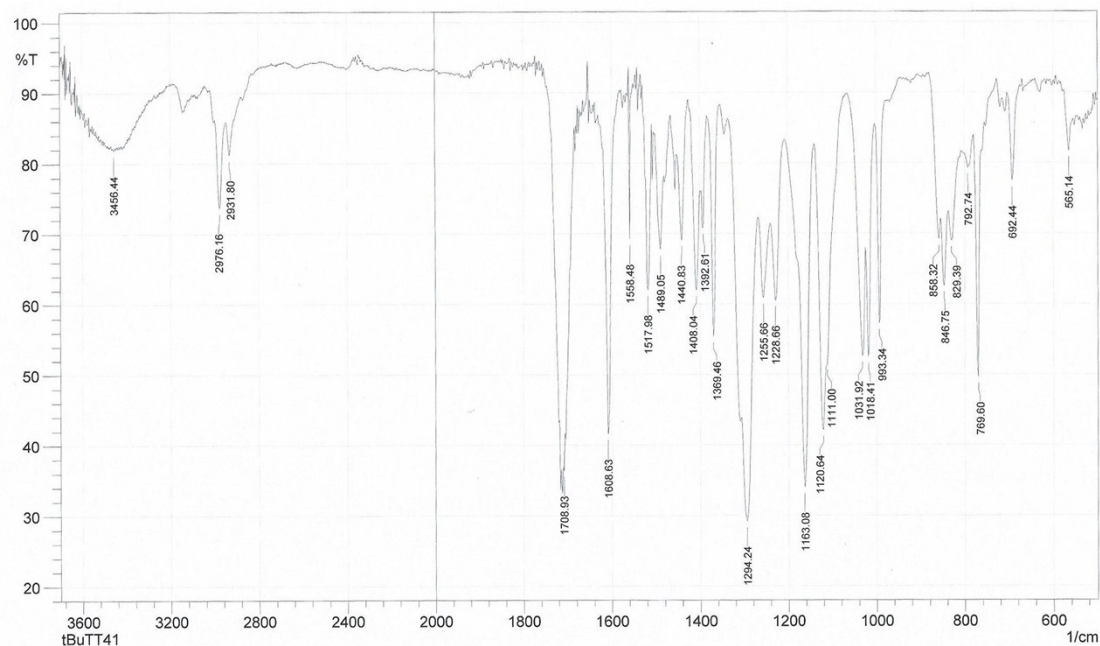
Elements Used:
 C: 76-78 H: 70-75 N: 12-12 O: 6-9
 C77H72N12O8
 Ipz-1 11 (0.258)

1: TOF MS ES+
 5.06e+000

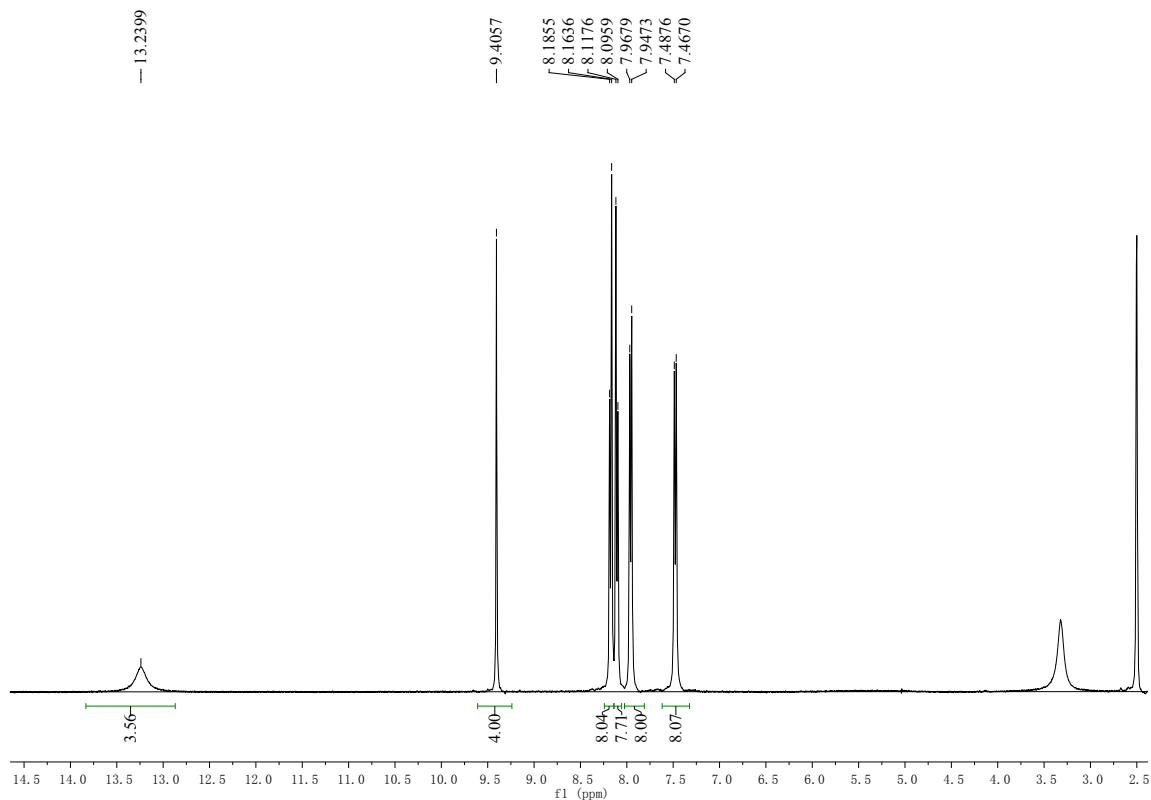


Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
1293.5679	1293.5674	0.5	0.4	47.5	13.2	0.0	C77 H73 N12 O8

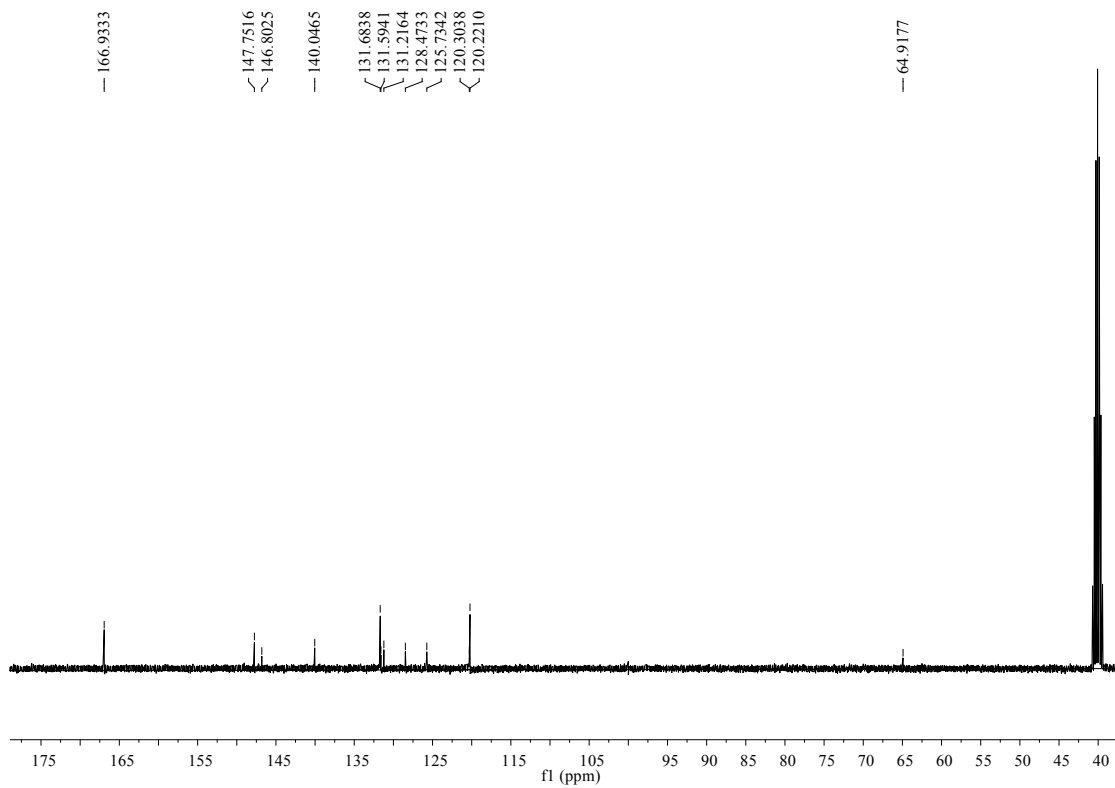
HR-MS spectrum of tetra-(*tert*-butyl) 4,4',4'',4'''-((methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1*H*- 1,2,3-triazole-4,1-diyl))tetrabenzoate.



FT-IR spectrum of tetra-(*tert*-butyl) 4,4',4'',4'''-((methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1*H*- 1,2,3-triazole-4,1-diyl))tetrabenzoate.



^1H NMR spectrum of 4,4',4'',4'''-((Methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1H-1,2,3-tri-azole-4,1-diyl))tetrabenzoic acid in $\text{DMSO-}d_6$.



^{13}C NMR spectrum of 4,4',4'',4'''-((Methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1H-1,2,3-tri-azole-4,1-diyl))tetrabenzoic acid in $\text{DMSO-}d_6$.

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions

1 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

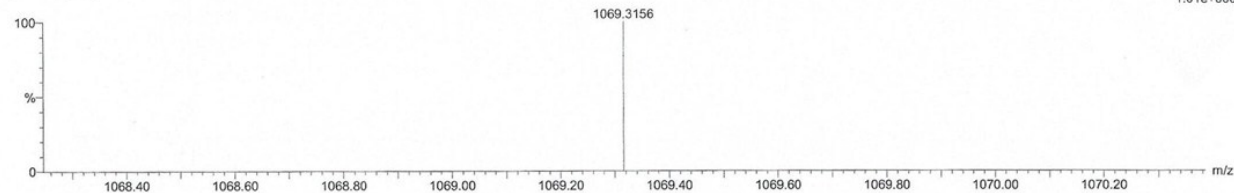
Elements Used:

C: 61-61 H: 40-42 N: 12-12 O: 6-9

C77H72N12O8

lpz-1 17 (0.397)

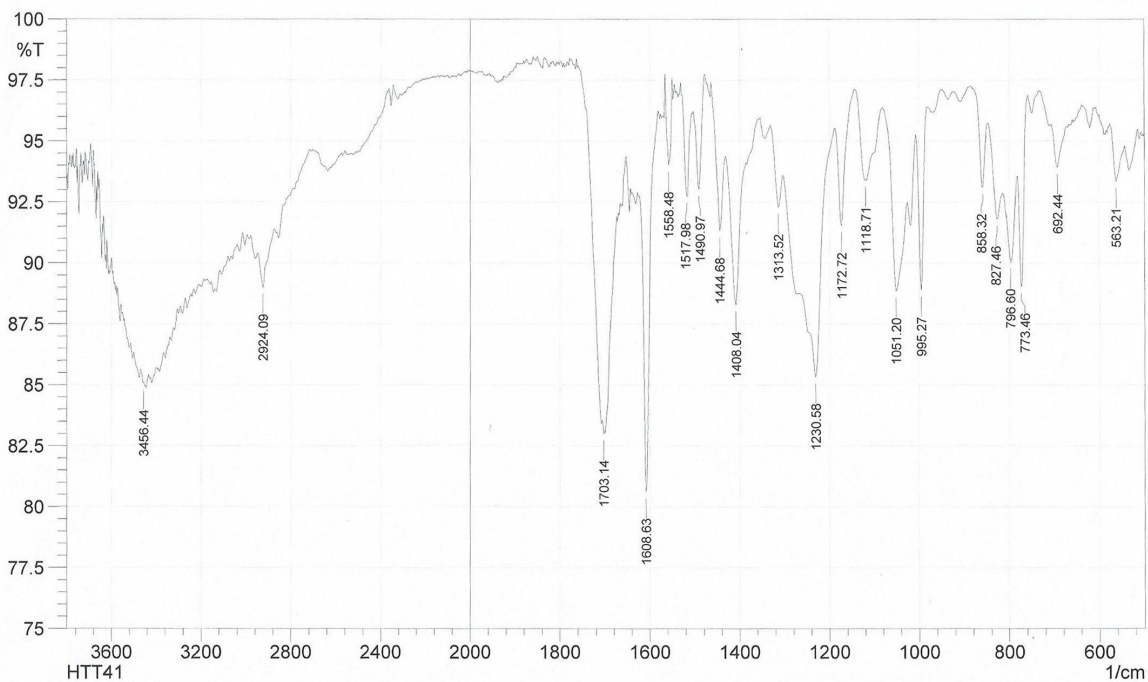
1: TOF MS ES+
1.01e+000



Minimum: -1.5
 Maximum: 5.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
1069.3156	1069.3170	-1.4	-1.3	47.5	12.1	0.0	C61 H41 N12 O8

HR-MS spectrum of 4,4',4'',4'''-((Methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1*H*-1,2,3-tri-azole-4,1-diyl))tetrabenzoic acid.



FT-IR spectrum of 4,4',4'',4'''-((Methanetetrayltetrakis(benzene-4,1-diyl)) tetrakis (1*H*-1,2,3-tri-azole-4,1-diyl))tetrabenzoic acid.

References

- S1. P.-Z. Li, X.-J. Wang, S. Y. Tan, C. Y. Ang, H.-Z. Chen, J. Liu, R.-Q. Zou and Y. Zhao, *Angew. Chem. Int. Ed.*, 2015, **54**, 12748.
- S2. P.-Z. Li, X.-J. Wang, J. Liu, J. S. Lim, R. Zou and Y. Zhao, *J. Am. Chem. Soc.*, 2016, **138**, 2142.
- S3. O. Plietzsch, C. I. Schilling, M. Tolev, M. Nieger, C. Richert, T. Muller and S. Brase, *Org. Biomol. Chem.*, 2009, **7**, 4734.
- S4. CrysAlisPro, Agilent Technologies, Version 1.171.36.28 (release 01-02-2013 CrysAlis171 .NET).
- S5. G. M. Sheldrick, *Acta Cryst.*, 2015, **C71**, 3-8.
- S6. A. L. Spek, *Acta Cryst.*, 2015, **C71**, 9-18.